APPLICATION No.: ATTORNEY DOCKET No.: CL 1943 US NA

10/630,248

PATENT **GROUP ART UNIT 1762**

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE APPLICATION OF:

XUEYING HUANG, ET AL.

APPLICATION NO.:

10/630,248

1762

FILED:

EXAMINER:

JULY 30, 2003

JIMMY LIN

FOR:

ATTORNEY DOCKET NO .:

MICROPARTICLE-BASED METHODS AND

SYSTEMS AND APPLICATIONS THEREOF

CL 1943 US NA

GROUP ART UNIT:

DECLARATION UNDER 37 C.F.R. § 1.131

COMMISSIONER FOR PATENTS P.O. Box 1450 ALEXANDRIA, VA 22313-1450

Sir:

- I Ming Zheng, am a co-inventor with Dr. Xueying Huang¹ in the above-identified 1. patent application.
- I obtained a Bachelor's degree in Electronics from Peking University, People's 2. Republic of China in 1984 and a Ph.D. in Chemistry from Princeton University in 1995. I was a post-doctoral fellow at the National Institute of Health from 1996 to 2000.
- 3. I am currently employed by the Central Research and Development Department (hereinafter "CR & D") of E. I. du Pont de Nemours & Co., Wilmington, DE, United States of America (hereinafter "DuPont"), as a Research Associate. I joined DuPont in 2000. DuPont is the assignee of the above-referenced patent application.

¹ See accompanying declaration from Dr. Xueying Huang, also under 37 C.F.R. § 1.131.

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4. While working at C R & D of DuPont, in relation to the above-referenced patent application and otherwise, I used gold nanoparticles coated with ethylene glycol oligomer, and gold nanoparticles coated with a mixture of ethylene glycol and other ligands, that were made by Dr. Xueying Huang, to examine protein binding specificities. Said gold nanoparticles were prepared by Dr. Xueying Huang in 2001-2002, also working at DuPont CR &D, in Wilmington, DE, USA, at that time. Dr. Xueying Huang is no longer employed by DuPont.

- 5. In the Non-Final Office Action mailed on July 03, 2006, and subsequently in the Non-Final Office Action mailed on June 07, 2007 the Examiner rejected Claims 2, 5-17, and 19 under 35 U.S.C. § 103(a) as being obvious over Templeton, *et al.*, Langmuir 15:66-76 (1999), in view of Foos, *et al.*, Chem. Mater. 14:2401-08 (2002). Specifically, the Examiner asserted that "it would have been obvious to one of ordinary skill in the art at the time of the invention to have used an ethylene glycol oligomer in the preparation of water-soluble gold nanoparticles of Templeton because Foos teaches that an ethylene glycol oligomer can increase the water solubility of a gold nanoparticle."
- 6. I, Ming Zheng, declare that in September of 2001, at CR & D of DuPont in the United States, Dr. Xueying Huang and I reduced to practice the following entity:

water-soluble, metallic nanoparticles having a mixed monolayer of

- (i) a capture-coating component and
- (ii) a shielding component;

which was prior to the online publication date of Foos, *et al.* (April 19, 2002). Further to this declaration, I attach notebook pages signed by Dr. Xueying Huang and witnessed by co-workers as Exhibits 3H-8H, wherein the dates have been redacted. Also attached are pages from my notebooks (Exhibits 1 & 2), which have been signed by me and witnessed by my co-workers at DuPont.

Exhibit 1 exemplifies the reduction to practice of water-soluble, metallic nanoparticles made through Ligand Exchange reactions. For example, the data in the lower half of the page show two gel shift assays indicating the absence of non-specific protein binding on to ethylene glycol (abbreviated as "EG-SH" in the notebook page) coated Au particles. A protein called GST (noted by the gene name "pET41a" in the notebook page) was used for the assay. This protein has a GSH binding domain. When mixed with GSH coated Au particles ("Au-GSH"), we observed band shift as shown by lanes "1" in both gel images. When the GSH was exchanged by EG-SH, such band shift disappeared, as shown by lanes "2" in the "4% TBE" gel image, and lanes "2" and "3" in the "1% TBE" gel image. These data indicate that EG acts as an efficient shielding component.

Similarly, Exhibit 2 exemplifies the reduction to practice of water-soluble, metallic nanoparticles made through the ligand exchange reactions. In the previously submitted declaration under 37 C.F.R. § 131, dated December 03, 2006, I, Ming Zheng had erroneously stated that Exhibit 2 exemplifies nanoparticles made through <u>direct synthesis</u> reaction. By way of the present declaration, I, Ming Zheng hereby correct said erroneous statement. The correct statement is that Exhibit 2 exemplifies the reduction to practice of water-soluble, metallic nanoparticles made through the <u>ligand exchange</u> reactions. Particularly, the data in the upper left gel image show that a mixed monolayer with both EG and GSH at the ratio of 1: 6 provides specific binding (band shift with GST protein), yet resists non-specific binding (no band shift with BSA and streptavidin).

Although Exhibits 1 and 2 do not explicitly demonstrate that the final concentration of water in the reaction mixture for the direct synthesis of Au particles with ethylene glycol coating is from about 9% to about 18% V/V, as required by independent Claim 2, as suggested in the "Conclusions" of Exhibit 7H, it is clear that Dr. Xueying Huang and I were cognizant of the importance of the concentration of water to the stability of gold nanoparticles. Particularly, the "Conclusions" (See bottom of notebook Page Exhibit 7H) in Exhibit 7H, which is

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dated February 25, 2002, i.e., prior to the effective date of the Foos reference, states that:

 Without CH₃COOH, control of NaBH₄ could lead to Au~~~EG₄ (a few drops of NaBH₄ solution) nanoparticles (purple). It is not stable in H₂O. After days (5~10), some ppt was formed.

With CH₃COOH, control of NaBH₄ is still needed.
 pH: 2.0→ 5.0? More NaBH₄ could be tolerated in the formation of Au~~~EG nanoparticles.
 Stability?

The absence of CH₃COOH in the first conclusion (i.e., higher concentration of water) and presence of CH₃COOH in the second conclusion (i.e., lower concentration of water) and Xueying Huang's comments about stability indicate his cognizance of the importance of the concentration of water to the stability and yield of the gold nanoparticles with ethylene glycol coating.

The concentration range of 9-18% V/V of water is only a preferred range of water concentration for direct synthesis. Secondly, the approach in Foos relates to ligand exchange. Foos does not relate to the direct synthesis Method. Moreover, Foos does not disclose or discuss the water concentration range or its importance in stability of gold nanoparticles. In fact, Foos relates to ligand exchange reactions, and the water content and its implications are relevant only in direct synthesis method.

The direct synthesis of gold particles coated with ethylene glycol, and ethylene glycol mixed with other ligands was developed and optimized over some period of time, starting no later than November 7, 2001 (see Exhibit 3H, line in the middle of the page), and with first sign of success around February 25, 2002 (See Exhibit 4H, Exhibit 5H TEM image of the Au(EG)₄ particle, and Exhibit 6H-8H on exploring conditions for stable particle formation).

7. I, Ming Zheng, also declare that although Exhibits 1, 2, 3H-8H, demonstrate the reduction to practice of the present invention for representative coated metallic nanoparticles, I believe that Dr. Xueying Huang and I have demonstrated

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reduction to practice for the claimed coated metallic nanoparticles because (i) the binding specificity and (ii) the resistance to non-specific binding are rendered by the choice of ligand and the function of ethylene glycol oligomers. The chemical identity of the core metal does not play a role here because the metal core is buried or shielded by the coating and does not interact directly with the environment.

As a person signing below:

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true. I also declare that all statements were made with knowledge that willful false statements, and the like, are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and any such willful false statements may jeopardize the validity of either the patent application or any patent issuing thereon.

Ming

Respectfully Submitted,

9/7/07

PATENT

Ming Zheng Date

EXHIBIT 1

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2. An-634 +EG-SH in M20 3				e e e e e e e e e e e e e e e e e e e	- v, va. 44.	
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EXHIBIT 2

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a a ry a magnificant (A) re	1% TBE, 90 v , 32 min	1% TBE, 90 v , 32 min		
	1, 2 ul of 11/14/01, Au-EG/GSH 1:5			
Manual Control of	2. 1+6 ul PET41a (GST)	1. 1 ul of 9/11/01, Au-E6 2. 1+ 6 ul BSA	G/GSH 1/6, 40-50% fraction	
NAC ASSUM SAFETY .	3. 1+ 6 ul PED107-6 (GST-ZFP) 4. 2 ul of 11/14/01, Au-EG	2. 1+ 6 ul Lysozyme		
TOPE MODULES 4	5. 1+ 6 ul PET41a (GST)	4. 1+ 6ul Streptavadin	n	
wastermounced to	6. 1+ 6 ul PED107-6 (GST-ZFP)	5. 1+ 6 ul PET41a (GST 6. 1+ 6 ul PED107-6 (G) ST-ZFP)	
	[protein] ~ 1 mg/ml	7. 1+ 6 al PED107-6 (10	0/22/01's , ~0.2 mg/ml)	
	-	8. 1+6 ul PED107-6 (10	0/22/01's, dialyzed, <0.2 mg/ml)	
		All other proteins ~ 1 m	g/ml	
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EXHIBIT 3H

TITLE	DATE
E 101228- 10 PURPOSE	
Suggestions and Ideas	
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Synthesis of a neutral surface	
MANCIA + MS ~ONO + GSH	Broad size distribution
	needs to be nurrowed.
15 -5	method: non-poter solvent
(Au) GSH	HZO/MOH
-0~0~5	4-20/E20H
	tho / Acetone
	H-O/ProH
	•
	i salas a
@ Direct synthesi's ? Tried before no	or working.
6) Replacement reaction?	
How to quantify the number of G	2 H 3
TP -	Further Separation
TP (Au) + HS~ + GSH	
TP TP	(TP)n
Ratio: 50=1	
25:1	(An) (GSH) 2 logards,
	(18) a suchas
	(Bu) + GSM) z Protein,
1	DWA,
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EXHIBIT 4H

	synthesis of An	<u> </u>		E 101228- 57
reaction:		LIE O M	/Acelos and	
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EXHIBIT 5H

TITLE Bibelectronies	DATE	MAGARAMATA
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EXHIBIT 6H

TITLE Broelect	. ~	DATE	Marie 101000 Ho
	ynthesis of An	· 694	E 101228- 59
Recipe:			
H20 (nanopi	ive) 14 ml		
Chtzcoom .	0,5mL		
HANCIG	<u> </u>	(o.zmmol)	the holes were the holes when the holes were the holes were the holes when the holes were the holes were the holes when the holes were the holes when the holes were the holes were the holes when the holes were the holes were the holes when the holes were the holes when the holes were the holes were the holes when the holes when the holes were the holes when the ho
EGH-SH (10W	13/ml) 28.4mg	(o.Immol)	
NABHU	0.15		
H-20	5.0 mL	······································	
	2	added wast-ty sol	ubian:
	A Assessment of the second of	added wasteral	many bubbles
	Jil Lot	OUZINA.	were formed.
	() 3		
Lms	1001	# 4	
Fretons	#2 #3	O _i	
			And the second s
			· · · · · · · · · · · · · · · · · · ·
After a few drops	1357ec 6-3		odded ~ 2.0 ml
out the beginning,	drops, took	NABHH added	NaBH4.
took ~ o.5ml	no.5ml solution.	took ~ o.sml	color as grey.
solution, the solution was clear		Solution. I clear & purple.	After 1 day, the
purple.	tastall stable	After I day mater	is easy to sink do
However, it grad		Sink to the bottom	. 5
changed into grey a		sample vial.	
ppt was formed.			1
er ern ern er ern erne ern ern ern ern e			
The state of the s			
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EXPERIMENTER V	you When a	DATE	
MUTAIFOOFF SW			· videlfurframmen na senova rendermentaleus accion
WITNESSED BY . (🛩 🥒	1	DATE	<u></u>

EXHIBIT 7H

TITLE Bioelectronics DATE
E 101228- 60 PURPOSE Direct Synthesis of And—EGu
Recipe:
H20/CH3COUH/GG4-SH
refuctue NaBMu/H2D
1440 5000 1100
CATZODA ON WE ACRES
HAUCIU 0.0253
Equ-SH 5.0mg
NaBH 4 0.035 9
H20 2.0g
Ofter ~1.0 ml washy was added, the solution became grey.
m 10+ of lumbbles were formed.
420 5.05 7 na ~
CHOCOSH 0.5 Mi) /" = 2.0
- No Edu Sh
MANCHY 0.0255) When NUBITY WAS added, ppt was
and immediately.
H20 2.09 Did not work
Conclusions: (5 without Chacoom, Control of Norma could lead to (An) 1564
(a few drops of NaBHa solution): nanoparticles (purple).
It is not stable in H2O.
rofter stays (5~10). some ppt was formed.
(2) With CHIZCOOH, control of NABHLY is still needed.
gration of An-EGH nanoparticles.
Stability >
2. WOLLIA 1
EXPERIMENTER Query DATE
WITNESSED BY DATE

EXHIBIT 8H